

EXHIBIT C

CHAIN-OF-CUSTODY FORMS



CITY OF SAN DIEGO
METROPOLITAN WASTEWATER DEPARTMENT
ENVIRONMENTAL MONITORING & TECHNICAL SERVICES DIVISION
WASTEWATER CHEMISTRY LABORATORY
5530 Kiowa Drive
La Mesa, CA 91942
(619) 668-3215



SAMPLING REPORT & CHAIN OF CUSTODY RECORD – DAILY SAMPLING (BOD/SBOD)

Project/Client: Brown and Caldwell / EPMD	Sampler/s:	Type of Sampling Equipment/How sample obtained/other sampling notes: ISCO Autosampler – Time Composited
Contact Name: Victor Occiano	Contact Name: Joe Cordova	
Phone/Fax: (619) 203-3077 / (858) 514-8833	Phone: 619-221-8728	

Sample Information: (All information is required) Number of attachments: _____

Date/Time Sample Taken	Sampler	Source / Location	Sample Type/Description	Grab / Composite	Total vol/wt mLs / Gms	Number of containers	Container Type	Preservativ e	Analyses requested	Sample Log Number (Lab use only)
		Influent/ 13-Inf	Daily	Composite		1	P	4 °C	BOD ₅ , SBOD ₅ , COD, TSS, VSS, TKN, NH ₃ -N, Ort-P, Alkalinity	
		Biostyr Effluent / 13-BSEff	Daily	Composite		1	P	4 °C	BOD ₅ , SBOD ₅ , COD, TSS, VSS, TKN, NH ₃ -N, Ort-P, Alkalinity	
		Biofor-C Effluent / 13-BFCEff	Daily	Composite		1	P	4 °C	BOD ₅ , SBOD ₅ , COD, TSS, VSS, TKN, NH ₃ -N, Ort-P, Alkalinity	
		Influent/ 13-Inf-2	Daily	Composite		1	P	4 °C	BOD ₅ , SBOD ₅ , COD, TSS, VSS, TKN, NH ₃ -N, Ort-P, Alkalinity	
		Densadeg Influent / 3-DInf	Daily	Composite		1	P	4 °C	BOD ₅ , SBOD ₅ , COD, TSS, VSS, TKN, NH ₃ -N, Ort-P, Alkalinity	
		Densadeg Sludge / 3-DS	Daily	Grab		1	P		TV, VS	
		Biofor-N Effluent / 13-BFNEff	Daily	Composite		1	P	4 °C	BOD ₅ , SBOD ₅ , COD, TSS, VSS, TKN, NH ₃ -N, Ort-P, Alkalinity	

Chain-of-Custody

Comments

Relinquished by: Name:	Received by: Name:	Date & Time		
Sign:	Sign:	Location:		
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			Reviewed by:	Date:



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La Mesa, CA 91942
(619) 668-3215



SAMPLING REPORT & CHAIN OF CUSTODY RECORD – DAILY SAMPLING (CBOD/SCBOD)

Project/Client: Brown and Caldwell / EPMD Contact Name: Victor Occiano Phone/Fax: (619) 203-3077 / (858) 514-8833	Sampler/s: Contact Name: Joe Cordova Phone: 619-221-8728	Type of Sampling Equipment/How sample obtained/other sampling notes: ISCO Autosampler – Time Composited
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Sample Information: (All information is required) Number of attachments: _____

Date/Time Sample Taken	Sampler	Source / Location	Sample Type/Description	Grab / Composite	Total vol/wt mLs / Gms	Number of containers	Container Type	Preservative	Analyses requested	Sample Log Number (Lab use only)
		Influent/ 13-Inf	Daily	Composite		1	P	4 °C	CBOD ₅ , SCBOD ₅ , COD, TSS, VSS, TKN, NH ₃ -N, Ort- P, Alkalinity	
		Biostyr Effluent / 13-BSEff	Daily	Composite		1	P	4 °C	CBOD ₅ , SCBOD ₅ , COD, TSS, VSS, TKN, NH ₃ -N, Ort- P, Alkalinity	
		Biofor-C Effluent / 13-BFCEff	Daily	Composite		1	P	4 °C	CBOD ₅ , SCBOD ₅ , COD, TSS, VSS, TKN, NH ₃ -N, Ort- P, Alkalinity	
		Influent/ 13-Inf-2	Daily	Composite		1	P	4 °C	CBOD ₅ , SCBOD ₅ , COD, TSS, VSS, TKN, NH ₃ -N, Ort- P, Alkalinity	
		Densadeg Influent / 3-DInf	Daily	Composite		1	P	4 °C	CBOD ₅ , SCBOD ₅ , COD, TSS, VSS, TKN, NH ₃ -N, Ort- P, Alkalinity	
		Densadeg Sludge / 3-DS	Daily	Grab		1	P		TV, VS	
		Biofor-N Effluent / 13-BFNEff	Daily	Composite		1	P	4 °C	CBOD ₅ , SCBOD ₅ , COD, TSS, VSS, TKN, NH ₃ -N, Ort- P, Alkalinity	

Chain-of-Custody

Comments

Relinquished by: Name: Sign:	Received by: Name: Sign:	Date & Time Location:	
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SAMPLING REPORT & CHAIN OF CUSTODY RECORD – EVERY OTHER DAY (BACKWASH SAMPLES)

Project/Client:	Brown and Caldwell / EPMD	Sampler/s:		Type of Sampling Equipment/How sample obtained/other sampling notes:
Contact Name:	Victor Occiano	Contact Name:	Joe Cordova	Grab Sample from Backwash Tank/Recycle Pump
Phone/Fax:	(619) 203-3077 / (858) 514-8833	Phone:	619-221-8728	

Sample Information: (All information is required) Number of attachments: _____

Date/Time Sample Taken	Sampler	Source / Location	Sample Type/Description	Grab / Composite	Total vol/wt mLs / Gms	Number of containers	Container Type	Preservative	Analyses requested	Sample Log Number (Lab use only)
		Biostyr Backwash/ 13-BSBW	Every Other Day	Grab		1	P	4 °C	BOD ₅ , SBOD ₅ , COD, TSS, VSS, TS, VS, SETS	
		Biofor-C Backwash / 13-BFCBW	Every Other Day	Grab		1	P	4 °C	BOD ₅ , SBOD ₅ , COD, TSS, VSS, TS, VS, SETS	
		Biofor-N Backwash / 13-BFNBW	Every Other Day	Grab		1	P	4 °C	BOD ₅ , SBOD ₅ , COD, TSS, VSS, TS, VS, SETS	
		Combined Backwash/ 3-CBW	Every Other Day	Grab		1	P	4 °C	BOD ₅ , SBOD ₅ , COD, TSS, VSS, TS, VS, SETS	

Chain-of-Custody

Comments

Relinquished by: Name:	Received by: Name:	Date & Time		
Sign:	Sign:	Location:		
Relinquished by:	Received by:	Date & Time		
Name:	Name:	Location:		
Sign:	Sign:			
Relinquished by:	Received by:	Date & Time		
Name:	Name:	Location:		
Sign:	Sign:			
			Reviewed by:	Date:

See instructions, on reverse, for completing this form.

FIGURE 2a

Original - retained by Lab.1st copy - Transporter
Last copy - for sample originator



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La Mesa, CA 91942
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SAMPLING REPORT & CHAIN OF CUSTODY RECORD – IMHOFF CONE ANALYSIS

Project/Client:	Brown and Caldwell / EPMD	Sampler/s:		Type of Sampling Equipment/How sample obtained/other sampling notes:
Contact Name:	Victor Occiano	Contact Name:	Joe Cordova	Grab Sample from Backwash Tank/Recycle Pump
Phone/Fax:	(619) 203-3077 / (858) 514-8833	Phone:	619-221-8728	

Sample Information: (All information is required) Number of attachments: _____

Date/Time Sample Taken	Sampler	Source / Location	Sample Type/Description	Grab / Composite	Total vol/wt mLs / Gms	Number of containers	Container Type	Preservative	Analyses requested	Sample Log Number (Lab use only)
		Biostyr Backwash/ 13-BSBW	Supernatant	Grab		1	P		TSS, VSS, CBOD	
		Biostyr Backwash/ 13-BSBW	Solids	Grab		1	P		TS, VS	
		Biofor-C Backwash / 13-BFCBW	Supernatant	Grab		1	P		TSS, VSS, CBOD	
		Biofor-C Backwash / 13-BFCBW	Solids	Grab		1	P		TS, VS	
		Combined Backwash/ 3-CBW	Supernatant	Grab		1	P		TSS, VSS, CBOD	
		Combined Backwash/ 3-CBW	Solids	Grab		1	P		TS, VS	

Chain-of-Custody

Comments

Relinquished by: Name: Sign:	Received by: Name: Sign:	Date & Time Location:		
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Relinquished by: Name: Sign:	Received by: Name: Sign:	Date & Time Location:		
			Reviewed by:	Date:

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La Mesa, CA 91942
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SAMPLING REPORT & CHAIN OF CUSTODY RECORD – BACTERIA SAMPLE

ANALYSIS

Project/Client:	Brown and Caldwell / EPMD	Sampler/s:	Smoczynski/ Suhendra	Type of Sampling Equipment/How sample obtained/other sampling notes:
Contact Name:	Victor Occiano	Contact Name:	Brent Bowman	MS2 Phage, Enterococcus, Total and Fecal Coliform for BAF Pilot Study
Phone/Fax:	(619) 203-3077 / (858) 514-8833	Phone:	619-221-8765	

Sample Information: (All information is required) Number of attachments: _____

Date/Time Sample Taken	Sampler	Source / Location	Sample Type/Description Grab / Composite	Total vol/wt mLs / Gms	Number of containers	Container Type	Preservative	Analyses requested	Sample Log Number (Lab use only)
	Smoczynski/ Suhendra	13-BFCEFF	Grab Sample	250	1	P	Blue Ice	MS2 phage, Enterococcus, Total and Fecal Coliform	
	Smoczynski/ Suhendra	13-BFNEFF	Grab Sample	250	1	P	Blue Ice	MS2 phage, Enterococcus, Total and Fecal Coliform	
	Smoczynski/ Suhendra	13-BSEFF	Grab Sample	250	1	P	Blue Ice	MS2 phage, Enterococcus, Total and Fecal Coliform	
	Smoczynski/ Suhendra	13-Inf	Grab Sample	250	1	P	Blue Ice	MS2 phage, Enterococcus, Total and Fecal Coliform	

Chain-of-Custody

Comments

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SAMPLING REPORT & CHAIN OF CUSTODY RECORD – COLLIMATED BEAM

SAMPLE

Project/Client:	Brown and Caldwell / EPMD	Sampler/s:	Smoczynski/ Suhendra	Type of Sampling Equipment/How sample obtained/other sampling notes:
Contact Name:	Victor Occiano	Contact Name:	Brent Bowman	
Phone/Fax:	(619) 203-3077 / (858) 514-8833	Phone:	619-221-8765	

Sample Information: (All information is required) Number of attachments: _____

Date/Time Sample Taken	Sampler	Source / Location	Sample Type/Description Grab / Composite	Total vol/wt mLs / Gms	Number of containers	Container Type	Preservative	Analyses requested	Sample Log Number (Lab use only)
	Smoczynski/ Suhendra	13-BFCEFF	Grab Sample	250	1	P	Blue Ice		
	Smoczynski/ Suhendra	13-BFNEFF	Grab Sample	250	1	P	Blue Ice		
	Smoczynski/ Suhendra	13-BSEFF	Grab Sample	250	1	P	Blue Ice		

Chain-of-Custody

Comments

Relinquished by: Name: Sign:	Received by: Name: Sign:	Date & Time Location:		
Relinquished by: Name: Sign:	Received by: Name: Sign:	Date & Time Location:		
Relinquished by: Name: Sign:	Received by: Name: Sign:	Date & Time Location:		
			Reviewed by:	Date:

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La Mesa, CA 91942
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SAMPLING REPORT & CHAIN OF CUSTODY RECORD – DAFT STUDY

Project/Client:	Brown and Caldwell / EPMD	Sampler/s:		Type of Sampling Equipment/How sample obtained/other sampling notes:
Contact Name:	Victor Occiano	Contact Name:	Brent Bowman	Note: Make sure DAFT influent water is also tested for CBOD ₅ , and TSS.
Phone/Fax:	(619) 203-3077 / (858) 514-8833	Phone:	619-221-8765	

Sample Information: (All information is required) Number of attachments: _____

Date/Time Sample Taken	Sampler	Source / Location	Sample Type/Description Grab / Composite	Total vol/wt mLs / Gms	Number of containers	Container Type	Preservative	Analyses requested	Sample Log Number (Lab use only)
		1-DAEff-	Grab Sample	1500	1	P	Blue Ice	CBOD ₅ , TSS	
		1-DAS-	Grab Sample	250	1	P	Blue Ice	TS, VS	
		2-DAEff-	Grab Sample	1500	1	P	Blue Ice	CBOD ₅ , TSS	
		2-DAS-	Grab Sample	250	1	P	Blue Ice	TS, VS	
		3-DAEff-	Grab Sample	1500	1	P	Blue Ice	CBOD ₅ , TSS	
		3-DAS-	Grab Sample	250	1	P	Blue Ice	TS, VS	

Chain-of-Custody

Comments

Relinquished by: Name: Sign:	Received by: Name: Sign:	Date & Time Location:		
Relinquished by: Name: Sign:	Received by: Name: Sign:	Date & Time Location:		
Relinquished by: Name: Sign:	Received by: Name: Sign:	Date & Time Location:		
			Reviewed by:	Date:

See instructions, on reverse, for completing this form.

FIGURE 2a

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SAMPLING REPORT & CHAIN OF CUSTODY RECORD – TKN SAMPLE ANALYSIS

Project/Client: Brown and Caldwell / EPMD	Sampler/s:	Type of Sampling Equipment/How sample obtained/other sampling notes:
Contact Name: Victor Occiano	Contact Name: Brent Bowman	TKN Samples for BAF Pilot Study
Phone/Fax: (619) 203-3077 / (858) 514-8833	Phone: 619-221-8765	

Sample Information: (All information is required) Number of attachments: _____

Date/Time Sample Taken	Sampler	Source / Location	Sample Type/Description	Grab / Composite	Total vol/wt mLs / Gms	Number of containers	Container Type	Preservative	Analyses requested	Sample Log Number (Lab use only)
		Influent/ 13-Inf	Daily	Composite		1	P	4 °C	TKN	
		Biostyr Effluent / 13-BSEff	Daily	Composite		1	P	4 °C	TKN	
		Biofor-C Effluent / 13-BFCEff	Daily	Composite		1	P	4 °C	TKN	
		Densadeg Influent/ 3-DInf	Daily	Composite		1	P	4 oC	TKN	
		Influent/ 13-Inf-2	Daily	Composite		1	P	4 °C	TKN	
		Biofor-N Effluent / 13-BFNEff	Daily	Composite		1	P	4 oC	TKN	
		Influent/ 13-Inf	Daily	Composite		1	P	4 °C	TKN	
		Biostyr Effluent / 13-BSEff	Daily	Composite		1	P	4 °C	TKN	
		Biofor-C Effluent / 13-BFCEff	Daily	Composite		1	P	4 °C	TKN	
		Densadeg Influent/ 3-DInf	Daily	Composite		1	P	4 oC	TKN	
		Influent/ 13-Inf-2	Daily	Composite		1	P	4 °C	TKN	
		Biofor-N Effluent / 13-BFNEff	Daily	Composite		1	P	4 °C	TKN	

Chain-of-Custody

Comments

Relinquished by: Name: Sign:	Received by: Name: Sign:	Date & Time Location:		
Relinquished by: Name: Sign:	Received by: Name: Sign:	Date & Time Location:		
Relinquished by: Name: Sign:	Received by: Name: Sign:	Date & Time Location:		
			Reviewed by:	Date:

See instructions, on reverse, for completing this form.

FIGURE 2a

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La Mesa, CA 91942
(619) 668-3215



SAMPLING REPORT & CHAIN OF CUSTODY RECORD – BIOMASS SAMPLING

Project/Client: Brown and Caldwell / EPMD	Sampler/s:	Type of Sampling Equipment/How sample obtained/other sampling notes:
Contact Name: Victor Occiano	Contact Name: Joe Cordova	BAF Columns
Phone/Fax: (619) 203-3077 / (858) 514-8833	Phone: 619-221-8728	

Sample Information: (All information is required) Number of attachments: _____

Date/Time Sample Taken	Sampler	Source / Location	Sample Type/Description	Grab / Composite	Total vol/wt mLs / Gms	Number of samples	Container Type	Preservative	Analyses requested	Sample Log Number (Lab use only)
		Biostyr	Biomass media	Grab	At least 15 piece				TSS, VSS	
		Biofor-C	Biomass media	Grab	At least 15 piece				TSS, VSS	

Chain-of-Custody

Comments

Relinquished by: Name: Sign:	Received by: Name: Sign:	Date & Time Location:		
Relinquished by: Name: Sign:	Received by: Name: Sign:	Date & Time Location:		
Relinquished by: Name: Sign:	Received by: Name: Sign:	Date & Time Location:		
			Reviewed by:	Date:

See instructions, on reverse, for completing this form.

FIGURE 2a

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**Instructions for completing the
SAMPLING REPORT & CHAIN-OF-CUSTODY RECORD**

FIGURE 2b

Prior to the acceptance into the Wastewater Chemistry Laboratory of any sample, this form will be completed by the sampler and submitting agency.

This form will be completed by the sampler at the time of sampling. Each person who takes custody of the sample must sign for acceptance (Received by:), and then again, upon relinquishing the sample to another person. The laboratory will sign for the acceptance and then provide a copy of this form to the person submitting the sample. The laboratory will maintain the originals of completed SAMPLING REPORT & CHAIN-OF-CUSTODY RECORDs in the laboratory.

The Sample information will be completed by the sampler/submitting agency in full. The following information is the minimum required and a column for this data is provided for on the form.

! Who is requesting the sample/analysis or what project the sample is for; include name and phone number.

! Who performed the sampling; include name and phone number.

! Any pertinent supporting information about the sampling, the sample, the location, weather, etc.

! How the samples was obtained, what sampling equipment used, and other information about the sampling event.

! Date and time of sampling.

! Source and location where the sample was taken, please provide full descriptive information.

Examples: Fiesta Is. Drying Bed #2; Fiesta Is. Dried Sludge Pile #16; PLR COMP, PLE COMP, Pt. Loma headworks;

! Sample type/description.

Examples: Decant/return stream; Dried Sludge; PLR-sewage influent Pt Loma;

! Total amount of sample, in mLs or Grams or other convenient measure.

! Number of sample containers. Should be the total for each sampling event/log number.

! Container Type; eg. 1L glass bottle, 5 gal plastic, etc.

! Preservative used; eg., HCl to pH <4, refeed, frozen, etc.

! Analysis/es requested; eg. BOD, Total Solids, % Moisture, Pesticides, Cyanides, etc.

! The SAMPLE LOG number is reserved for laboratory personnel who log the sample into the laboratory.

CHAIN-OF-CUSTODY

Sample chain-of-custody can be an important method to ensure that samples are properly identified, preserved, analyzed and reported. Also, the ability to document the possession of a sample throughout its analytical life may become a legal issue. Each person who takes possession and control of the sample will document the change of custody in the Chain-of-Custody part of the SAMPLING REPORT & CHAIN-OF-CUSTODY RECORD form. If you give the sample to someone (for example, to transport it to the lab.) you must complete the first available "Relinquished by" section and have the person you are giving the sample to complete the "Received by" section adjacent. Enter the date & time of the transaction in the Date & time block. Record where the transfer of custody took place in this block. The laboratory should provide the submitter with a copy of this form, retaining the original in lab. records.

COMMENTS

In addition to the previous Remarks section, this section is for comments concerning the sample, condition of sample upon transfer or receipt, delays in transfer, special instructions or other information relevant to the sample or sampling event. You are encouraged to make any comment that may have a bearing on the sample, its handling or its analysis.

EXHIBIT D

**COMPOSITE SAMPLING
INSTRUCTIONS**

Refer to Phase I Protocol

EXHIBIT E

DAILY PILOT TEST METER AND INSTRUMENTATION READINGS LOG SHEETS

City of San Diego MWW BAF Pilot Study Sampling Program
Brown and Caldwell Project 24901
Daily Pilot Test Unit Instrument & Meter Readings
@ NORMAL OPERATIONS

Location: 3DINF

Date:

Time:

Analyst:

PORTABLE METER READINGS – 3Dinf		OBSERVATIONS	
Parameter	Value		Value/Comment
Temperature, °C		Ferric Storage Leak Observed? Yes <input type="checkbox"/> No <input type="checkbox"/>	If yes to any, consult spill prevention plan and alert operations shift supervisor. Then call Victor Occiano @ (619) 203-3077 or Amer Barhoumi @ (619) 922-6421 and wait for instructions.
DO, mg/L		Ferric Tubing Leak Observed? Yes <input type="checkbox"/> No <input type="checkbox"/>	
pH reading of 7.0 Buffer		BAF Influent Tank Overflowing? Yes <input type="checkbox"/> No <input type="checkbox"/>	If no, call Victor Occiano @ (619) 203-3077 or Amer Barhoumi @ (619) 922-6421 and wait for instructions.
Calibrated pH slope (BC and EPMD only)		Influent Pump Operating? Yes <input type="checkbox"/> No <input type="checkbox"/>	If no, call Victor Occiano @ (619) 203-3077 or Amer Barhoumi @ (619) 922-6421 and wait for instructions.
pH		Influent Pump Primed? Water Flowing? Yes <input type="checkbox"/> No <input type="checkbox"/>	If no, call Victor Occiano @ (619) 203-3077 or Amer Barhoumi @ (619) 922-6421 and wait for instructions.
Turbidity, NTU		Provide Reason for pump failure	
UVT, %		Rapid mixing tank overflowing? Yes <input type="checkbox"/> No <input type="checkbox"/>	If yes, shake the butterfly valve to clean it. If it is still overflowing, call Victor Occiano @ (619) 203-3077 and wait for instructions.
Other Observations			

Location: DENSADEG

Date:

Time:

Analyst:

INSTRUMENT READINGS - DENSADEG			
Parameter		D_INF flow, gpm	
Pump speed of FeCl ₃ pump		Sludge Blanket Level (ft)	
Stroke number of FeCl ₃ pump		Blowdown Time (s) / Time Between Sludge Blowdowns (hr.)	
Pump speed of Polymer pump			
Stroke number of Polymer pump			
Calibration column:		PORTABLE METER READINGS –3DS	
FeCl ₃ , mL/min		pH	
Polymer, mL/min		Temperature, °C	
Other observations			

City of San Diego MWW BAF Pilot Study Sampling Program
Brown and Caldwell Project 24901
Daily Pilot Test Unit Instrument & Meter Readings
@ NORMAL OPERATIONS

Location: 13INF

Date:

Time:

Analyst:

PORTABLE METER READINGS - 13Inf		OBSERVATIONS	
Parameter	Value		Value/Comment
Temperature, °C		BAF Influent Tank Overflowing?	Yes <input type="checkbox"/> No <input type="checkbox"/> If no, check influent pump
DO, mg/L		Influent Pump Operating?	Yes <input type="checkbox"/> No <input type="checkbox"/> If no, call Victor Occiano @ (619) 203-3077 or Amer Barhoumi @ (619) 922-6421 and wait for instructions
pH reading of 7.0 Buffer		Influent Pump Primed? Water Flowing?	Yes <input type="checkbox"/> No <input type="checkbox"/> If no, call people listed above
Calibrated pH slope (BC and EPMD only)			
pH			
Turbidity, NTU		Provide Reason for pump failure	
UVT, %		Check DO Probe Membrane for Air Bubbles	Yes <input type="checkbox"/> No <input type="checkbox"/> If yes, notify Mitch Dornfeld, EPMD staff or BC staff to replace membrane
Other Observations			

Location: BIOSTYR Date:

Time:

Analyst:

INSTRUMENT READINGS - BIOSTYR		PORTABLE METER READINGS – 13BSEff	
Parameter	Value	Parameter	Value
Process Air Flow, scfm		pH	
Influent flow, gpm		Temperature, °C	
Backwash flow, gpm		DO, mg/L	
Effluent DO, mg/L		Turbidity, NTU	
Inlet Pressure, iwc		UVT, %	
Effluent pH, pH Unit			
Headloss, iwc		Effluent characteristics (visual, odor)	
Time till next BW, hr.			
Max time between BW, hr.			
Media loss	YES <input type="checkbox"/> NO <input type="checkbox"/> If Yes, what is the amount (1-4) where 1= 0-Few; 4= Severe Amount =		
Other observations			

City of San Diego MWW BAF Pilot Study Sampling Program
Brown and Caldwell Project 24901
Daily Pilot Test Unit Instrument & Meter Readings
@ NORMAL OPERATIONS

Location: BIOFOR C **Date:** **Time:** **Analyst:**

INSTRUMENT READINGS – BIOFOR C		PORTABLE METER READINGS – 13BFCEff	
Parameter	Value	Parameter	Value
Process Air Flow, scfm		pH	
Influent flow, gpm		Temperature, °C	
Column Pressure, psi		DO, mg/L	
		Turbidity, NTU	
Water Level in BIOFOR C Effluent Storage Tank Rising?	YES <input type="checkbox"/> NO <input type="checkbox"/> If no, adjust BIOFOR N Box Screen Valve	UVT, %	
BIOFOR N Box Screen Valve Adjustment; # Turns		Effluent characteristics (visual, odor)	
Media loss	YES <input type="checkbox"/> NO <input type="checkbox"/> If Yes, what is the amount (1-4) where 1= 0-Few; 4= Severe Amount =		
Condition of Screen (solids buildup, overflow, collected solids)			
Other observations			

Location: BIOFOR N **Date:** **Time:** **Analyst:**

INSTRUMENT READINGS – BIOFOR N		PORTABLE METER READINGS – 13BFNEff	
Parameter	Value	Parameter	Value
Process Air Flow, scfm		pH	
Influent flow, gpm		Temperature, °C	
Column Pressure, psi		DO, mg/L	
		Turbidity, NTU	
Effluent characteristics (visual, odor)		UVT, %	
Media loss	YES <input type="checkbox"/> NO <input type="checkbox"/> If Yes, what is the amount (1-4) where 1= 0-Few; 4= Severe Amount =		
Other observations			

City of San Diego MWW BAF Pilot Study Sampling Program

Brown and Caldwell Project 24901

Bi-Daily Pilot Test Unit Instrument Readings

DURING BACKWASH SAMPLING EVENT

Location: BioFor C **Date:** **Time:** **Analyst:**

INSTRUMENT READINGS – BIOFOR C BACKWASH EVENT	
Parameter	Value
Process Air Flow, scfm	
Influent Flow, gpm	
Backwash Air Flow, scfm	
Backwash Flow, gpm	
Inlet Pressure, psi	Pre BW= During BW= Post BW=
Headloss, iwc	Pre BW= Post BW=
Time till next BW, min.	
Max time between BW, min	
Backwash Tank Level, in	
Media loss	<div style="text-align: center;">- YES <input type="checkbox"/> NO <input type="checkbox"/> If Yes, what is the amount (1-4) where 1= 0-Few; 4= Severe Amount = </div>
Other observations	

Location: BioFor N **Date:** **Time:** **Analyst:**

INSTRUMENT READINGS – BIOFOR N BACKWASH EVENT	
Parameter	Value
Process Air Flow, scfm	
Influent Flow, gpm	
Backwash Air Flow, scfm	
Backwash Flow, gpm	
Inlet Pressure, psi	Pre BW= During BW= Post BW=
Headloss, iwc	Pre BW= Post BW=
Time till next BW, min.	
Max time between BW, min	
Backwash Tank Level, in.	
Media loss	<div style="text-align: center;">- YES <input type="checkbox"/> NO <input type="checkbox"/> If Yes, what is the amount (1-4) where 1= 0-Few; 4= Severe Amount = </div>
Other observations	

City of San Diego MWW BAF Pilot Study Sampling Program
Brown and Caldwell Project 24901
Bi-Daily Pilot Test Unit Instrument Readings
DURING BACKWASH SAMPLING EVENT

Location: BioStyr Date: Time: Analyst:

INSTRUMENT READINGS - BIOSTYR			
Parameter	Value		
Process Air Flow, scfm			
Influent flow, gpm			
Backwash Air Flow, scfm			
Backwash flow, gpm			
Effluent DO, mg/L			
Inlet Pressure, iwc			
Effluent pH, pH Unit			
Headloss, iwc	Pre BW=	During BW=	Post BW=
Time till next BW, hr.			
Max time between BW, hr			
Backwash Tank Level, in			
Media loss	<div style="display: flex; justify-content: space-between; align-items: flex-start;"> YES <input type="checkbox"/> NO <input type="checkbox"/> <div style="text-align: right;"> - If Yes, what is the amount (1-4) where 1= 0-Few; 4= Severe Amount = </div> </div>		
Other observations			

City of San Diego MWW D BAF Pilot Study Sampling Program
Brown and Caldwell Project 124901
Daily Pilot Test Unit Instrument & Meter Readings
@ NORMAL OPERATIONS

Date: Time: Analyst:

On-site Instrument Readings and Observations

Biofor-C Process Air	scfm
Biofor-C Influent Flow	gpm
Biofor-C Column Pressure	psi
Biostyr Process Air Flow	scfm
Biostyr Effluent D.O.	mg/L
Biostyr Inlet Pressure	iwc
Biostyr Effluent pH	
Biostyr Headloss	iwc
Biostyr Influent Flow	gpm

Liquid Phase Grab Sample Analysis – Readings and Observations

BAF Influent (13-INF)	
pH:	
Temp:	°C
Turbidity:	NTU
UVT:	%
Effluent Characteristics (Odor, clarity, solids, etc)	
BIOFOR-C Effluent (13-BFC EFF)	
pH:	
Temp:	°C
Turbidity:	NTU
UVT:	%
DO:	mg/L
Effluent Characteristics (Odor, clarity, solids, etc)	
BIOSTYR Effluent (13-BS EFF)	
pH:	
Temp:	°C
Turbidity:	NTU
UVT:	%
DO:	mg/L
Effluent Characteristics (Odor, clarity, solids, etc)	

City of San Diego MWW D BAF Pilot Study Sampling Program
Brown and Caldwell Project 24901
Imhoff Cone Testing

Location: BioFor C Date: Time: Analyst:

Minutes:	mL
15	
30	
Observations:	

Location: BioStyr Date: Time: Analyst:

Minutes:	mL
15	
30	
Observations:	

Sample: Combined BW Date: Time: Analyst:

Minutes:	mL
15	
30	
Observations:	

City of San Diego MWWD BAF Pilot Study Sampling Program
Brown and Caldwell Project 24901
Bench Scale DAFT Testing

Test: DAFT → Mixed BIOS TYR and BIOFOR C BW

Date: **Time:** **Batch #:** **Analyst:**

INSTRUMENT READINGS – DAFT Unit		PORTABLE METER READINGS –DAFT-Inf	
Parameter	Value	Parameter	Value
Volume of BW used, L		pH	
Polymer usage, mg/L		Temperature, °C	
Pressure applied, psi		Turbidity, NTU	
Duration of pressure application, min		PORTABLE METER READINGS –DAFT-Eff	
Sludge blanket level, in		pH	
Recycle ration used, %		Temperature, °C	
How fast is floatability of the solids?		Turbidity, NTU	
Other observations:			

City of San Diego MWW BAF Pilot Study Sampling Program
Brown and Caldwell Project 24901
Bench Scale DAFT Testing

Test: DAFT → CO-SETTLING

Date: Time: Batch #: Analyst:

INSTRUMENT READINGS – DAFT Unit		PORTABLE METER READINGS –DAFT-Inf	
Parameter	Value	Parameter	Value
Volume of sludge used, L		pH	
Polymer usage, mg/L		Temperature, °C	
Pressure applied, psi		Turbidity, NTU	
Duration of pressure application, min		PORTABLE METER READINGS –DAFT-Eff	
Sludge blanket level, in		pH	
Recycle ration used, %		Temperature, °C	
How fast is floatability of the solids?		Turbidity, NTU	
Other observations:			

EXHIBIT F

PRESSURE TRANSDUCER DATA DOWNLOAD INSTRUCTIONS

Refer to Phase I Protocol

EXHIBIT G

BACKWASH SAMPLING INSTRUCTIONS

Refer to Phase I Protocol

EXHIBIT H

SVI METHODOLOGY AND LOG SHEETS

Refer to Phase I Protocol

EXHIBIT I

STANDARD OPERATION PROCEDURE FOR BIOMASS MEDIA SAMPLING AND ANALYSIS

Refer to Phase I Protocol

EXHIBIT J

**CONTINUOUS TURBIDITY METER
LOG SHEETS**

Refer to Phase I Protocol

EXHIBIT K

OPERATING PROTOCOL FOR DAFT

City of San Diego MWW / Brown and Caldwell BAF Pilot Study – Phase II

Bench Scale DAFT Operating Procedure

In dissolved-air flotation (DAF) systems, air is dissolved in the wastewater under pressure of several atmospheres, followed by release of the pressure to the atmospheric level. Separation of solid particles from the liquid phase is brought by introducing fine gas bubbles into the liquid phase. The bubbles attach to the particulate matter, and the buoyant force of the combined particle and gas bubbles is great enough to cause the particle to rise to the surface. Particles that have a higher density than the liquid can thus be made to rise. Once the particles have been floated to the surface, they can be collected by a skimming operation. The principle advantage of flotation over sedimentation is that very small or light particles that settle slowly can be removed more completely and in a shorter time.

Picture of the bench scale DAF unit that is going to be used in this study is given in Figure K-1. The DAF unit footprint is roughly 2x3 feet and 3 feet high. An extra bench space is also needed for ancillary lab equipment. The DAF test apparatus consists of a pressure chamber, pressure gage, pressure release valve, high-pressure air source, a 1L-graduated cylinder, and under drain valves.

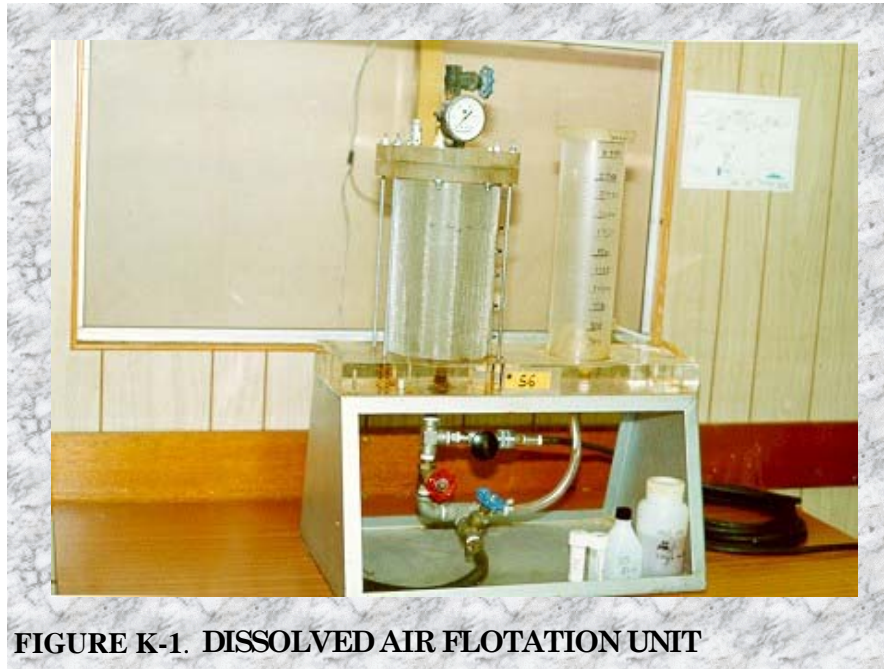


FIGURE K-1. DISSOLVED AIR FLOTATION UNIT

Test #1: Dedicated Thickening of BAF Backwash Water

Bench scale DAFT unit is a batch process, therefore several batches needs to be run for both Biostyr and Biofor-C backwash water. In these batches the aim is to optimize polymer dosage and recycle ratio in order to get the best effluent quality.

1. First of all, a Jar test should be applied to determine the optimum polymer dosage for the best flocculation. Cationic polymer will be used for the Jar test at 2, 5 and 10-mg/L concentration.
2. After determining the optimum polymer dosage, 4-L of backwash water sample will be mixed with the polymer in a container. After floc formation, the mixture will be poured slowly into the pressure chamber.
3. Pressure chamber will be capped tightly and pressure of 55 lb/in² will be applied with compressed air for 10-15 minutes to make sure that the wastewater is saturated with air.
4. Open the valve to transfer the pressurized wastewater to the 1-L graduated cylinder where air comes out of solution in minute bubbles throughout the entire volume of liquid. Bubbles make solids to float on the surface.
5. Meanwhile, do the necessary observations and fill out the DAFT log sheet given in Exhibit E.
6. After the wastewater in the graduated cylinder became calm, and the sludge float on the surface, decant the container by opening the drain valve. Collect the wastewater in a clean container. Close the drain valve before taking any sludge samples.
7. Collect the sludge sample into another clean container by either opening the drain valve or collect the sludge sample from the 1-L cylinder by the help of a small beaker.
8. Test the clear effluent for CBOD₅ and TSS. Test the sludge for TS and VS (%).

In most of the full-scale DAFT units, a portion of the DAF effluent (15 to 120%) is recycled, pressurized, and semi saturated with air. The recycled flow is mixed with the unpressurized main stream just before admission to the flotation tank. Pressurizing only the recycle stream is mainly used to reduce the cost of the DAF unit.

In this bench scale study one of the purposes is to try different recycle ratios and have an idea about the optimum recycle ratio. In order to test the effect of recycle on DAFT performance, a slight modification is needed in the procedures described above.

- R1. Save the DAF processed wastewater that is removed from the earlier batch run as described in step 5 above.
- R2. Mix this recycle effluent with the untreated backwash water to obtain the specified recycle ratio. Different recycle ratios will be tested for 15, 30, 50, 75, 100, and 120%.
- R3. Repeat steps 2 to 7 as described above. Use the optimum polymer dosage obtained in step 1 diluted by a factor at the recycle ratio being tested.

Once the optimum operation conditions found, the test will be repeated two more times to have test the repeatability of the result (i.e., step R3 is repeated twice for a total of 3 data points). Therefore, the effort involved in Test # 1 can be described as follows:

Jar testing	Occurs once using the 6-gang stirrer
Testing to find optimum recycle ratio	6 Runs
Repeated tests at optimum polymer and recycle	2 Runs

Test #2: Co-thickening of the Blended Primary and BAF Sludge

All the steps given in Test 1 will be repeated.

Test #3: Thickening of the Co-settled BAF Backwash and Primary Sludge

All the steps given in Test 1 will be repeated.

EXHIBIT L

LABORATORY PROCEDURES FOR FILTER FLOCCULATED BOD



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RAPID COMMUNICATION

A RAPID PHYSICAL-CHEMICAL METHOD FOR THE DETERMINATION OF READILY BIODEGRADABLE SOLUBLE COD IN MUNICIPAL WASTEWATER

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Abstract—A rapid physical-chemical method has been developed for the determination of the readily biodegradable portion of influent soluble COD. The method involves removal by flocculation and precipitation of colloidal matter that normally passes through 0.45 µm membrane filters. Results from four domestic wastewaters demonstrated that the physical-chemical method and the biological method (Ekama *et al.*, 1984) gave virtually identical results. The physical-chemical method was used successfully to measure the quantity of truly soluble organic matter removed in the anaerobic zone of bench-scale enhanced biological phosphorus removal activated sludge systems.

Key words—readily biodegradable COD, municipal wastewater, flocculation, activated sludge modeling

INTRODUCTION

The mathematical modeling of biological wastewater treatment processes and the design and operation of selector systems and nutrient removal plants require a reliable and accurate estimate of the readily biodegradable portion of influent wastewater COD (S_s). Readily biodegradable organic matter consists of simple organic molecules such as volatile fatty acids (VFA) and low molecular weight carbohydrates that can pass through the cell membrane and be metabolized within minutes (Henze *et al.*, 1987).

Currently, reliable measurements of S_s utilize a time-consuming biological method (Ekama *et al.*, 1984). The purpose of this paper is to present an alternative physical-chemical (flocculation) method for the determination of S_s . The validity of the method was assessed by comparing results obtained in parallel tests of the flocculation method and the biological method. In addition the method was successfully applied to continuous-flow enhanced biological phosphorus removal (EBPR) systems to determine the amount of truly soluble COD removed under anaerobic conditions.

RATIONALE OF FLOCCULATION METHOD

The flocculation method for determining S_s (S_{sfloc}) is based on the rationale that membrane filtration of a sample that has been flocculated (in this case by precipitating $Zn(OH)_2$ at pH 10.5) will produce a filtrate containing only truly soluble organic matter. The colloidal particles normally present in filtrates through membranes of 0.45 µm nominal pore diameter will be removed during the flocculation step preceding filtration. Ekama *et al.* (1984) and the IAWPRC Task Group on Activated Sludge Modeling (1986) proposed that influent S_s is related to the truly soluble influent COD by the equation:

$$S_s = COD_{sol} - Si \quad (1)$$

where

S_s = influent readily biodegradable soluble COD

COD_{sol} = influent total truly soluble COD

Si = influent non-readily biodegradable soluble COD.

The flocculation method determines COD_{sol} ($flocCOD_{sol}$) in equation (1). To derive a value of S_s from COD_{sol} , an estimate of Si must be obtained. Influent Si is considered equal to the truly soluble effluent COD of an activated sludge system treating the influent at an MCRT of greater than 3 days (Ekama *et al.*, 1984). When the wastewater of interest is already being treated in an activated sludge plant (laboratory pilot plant or full-scale) Si is determined by performing a $flocCOD_{sol}$ measurement on the effluent. When measuring $S_s(floc)$ for a wastewater for which a treatment plant does not exist, effluent Si is determined by performing a $flocCOD_{sol}$ analysis on the effluent of a 24hr fill-and-draw activated sludge system (MCRT > 3 days) fed with the wastewater of interest. This type of activated sludge system is much simpler to run than the continuous flow unit required for the biological method of determining S_s .

EXPERIMENTAL MATERIALS AND METHODS

Analytical Methods

Parallel S_s measurements were made by the biological and flocculation methods. Since the biological method is the currently accepted technique for measuring S_s it was used as the standard for assessing the validity of the flocculation method. A variety of domestic wastewaters containing S_s levels in the range 0-200 mg/l was tested. These samples included primary effluent from the Southeast (SE) and Richmond Sunset (RS) treatment plants in San Francisco, CA, raw City of Richmond, CA wastewater from Richmond Field Station (RFS), and primary effluent from the San Francisco Southeast plant supplemented with the centrate from an

Table 1: Average COD values of four types of wastewater tested, (mg/l)

COD value	SE Primary Effluent	RS Primary Effluent	RFS, Raw Wastewater	SE Primary Effluent + acid digester centrate
Influent soluble	122	105	92	185
Effluent soluble	50	61	51	69
Influent flocculated, CODsol	98.5	84	63	163
Effluent flocculated, Si	37	52	40.5	53
Influent, Ss	64.5	31.5	22	119
Influent, Ss(floc)	61.5	32	22.5	110

SE = San Francisco, CA, Southeast plant; RS = San Francisco, CA, Richmond Sunset plant; RFS = Richmond, CA Richmond Field Station

acid anaerobic sludge digester.

Chemical Oxygen Demand (COD)—All COD analyses were by the dichromate method Standard Methods, (1985), Section 508A.

Biological Method—A continuous-flow (3.4 l aeration basin, 1.4 l secondary clarifier) bench-scale activated sludge system was operated at an MCRT of 2.5 days in a semibatch mode (feed on 12 hr - feed off 12 hr). The value of Ss was estimated by measuring the step change in oxygen uptake rates (OUR) after the end of the feed period as described by Ekama *et al.* (1984). A heterotrophic yield value of 0.67 g biomass/g substrate (COD COD basis) was used (LAWPRC, 1986; Kappeler *et al.*, 1991).

Flocculation Method—Samples were flocculated by adding 1 ml of a 100 g/l zinc sulfate solution to a 100 ml wastewater sample and then mixing vigorously with a magnetic stirrer for approximately 1 min. The pH of the mixed sample was then adjusted to approximately 10.5 with 6 M sodium hydroxide solution and the sample allowed to settle quiescently for a few minutes. (Standard Methods, (1985), Section 417B). Clear supernatant (20-30 ml) was withdrawn with a pipette and passed through a 0.45 μ m membrane filter. The COD of the supernatant filtrate was determined. This COD was termed the flocculated soluble COD (flocCODsol). The coefficient of variation of flocCODsol measurements was 4%.

Laboratory Procedure

An acid anaerobic sludge digester was operated at a 4.0 day MCRT, an average pH of 5.8 and 37°C on a feed consisting of 40% primary and 60% waste activated sludge (TS basis) to provide effluent with a high readily biodegradable organic matter content. Average digester effluent VFA and CODsol concentrations were 2600 mg/l and 5900 mg/l respectively.

Two identical continuous-flow bench-scale EBPR activated sludge systems, immersed in water baths for temperature control were operated on settled Richmond, CA domestic wastewater supplemented with 50 mg/l sodium acetate as acetic acid (Mamais and Jenkins, 1992).

RESULTS AND DISCUSSION

Evaluation of flocculation method

The readily biodegradable COD content of wastewater samples was determined using both the biological and flocculation methods in parallel on 32 SE primary effluent samples, 14 RS primary effluent samples, 10 RFS raw wastewater samples and 11 SE primary effluent samples supplemented with acid anaerobic sludge digester centrate. Average values of influent and effluent CODsol and flocCODsol and Ss for the four types of wastewater tested are shown in Table 1. Biological and flocculation Ss values

were virtually identical for all wastewaters tested. SE primary effluent wastewater supplemented with acid anaerobic sludge digester centrate had the highest Ss due to its high VFA content; RFS raw wastewater taken during a rainy period had the lowest Ss values.

To demonstrate the equivalence of the two methods all Ss(floc) and Ss values obtained in this study are plotted against each other in Figure 1. The variables follow a linear relationship with a correlation coefficient of 0.965. Based on a linear regression analysis, assuming a zero intercept, Ss(floc) is equal to 1.025 Ss. A t-test carried out on the differences between each pair of measurements showed that both methods are identical at the 5% significance level (Chatfield, 1978).

Application of flocculation method to EBPR activated sludge systems

The effectiveness of the flocculation method for measuring Ss was tested using two continuous flow bench-scale

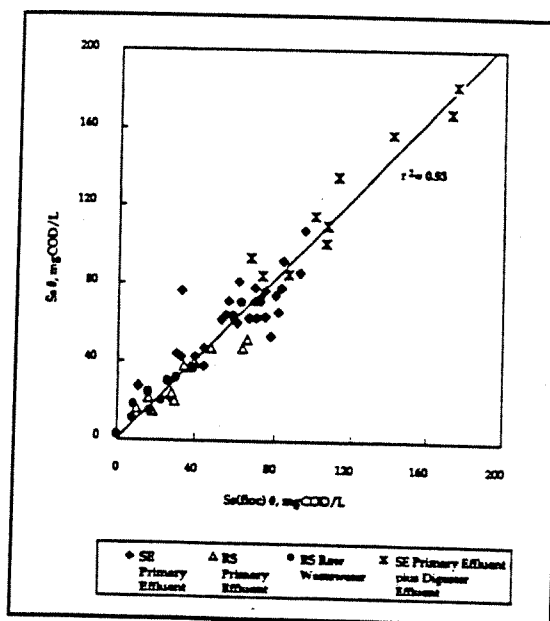


Fig. 1. Relationship of Ss(floc) to Ss for a variety of domestic wastewaters.

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Table 2. Average anaerobic zone soluble COD removal

Soluble COD Method	Anaerobic CODsol removal, mg/l		CODsol removed to Psol release ratio	
	EBPR*	EBPR*	EBPR*	EBPR*
Membrane filtration (CODsol)	55	74	28	5.3
Zn(OH) ₂ flocculation (flocCODsol)	4	29	2	2

*MCRT = 2.0 days; T = 20°C; Psol removal = 20%; TP in VSS = 2.7%
 *MCRT = 2.5 days; T = 20°C; Psol removal = 90%; TP in VSS = 6.9%

EBPR activated sludge systems under conditions of excellent and very poor EBPR. Influent and effluent soluble COD was measured using membrane filtration and the flocculation method. Soluble COD and soluble P (Psol) mass balances were conducted for the anaerobic zone and the CODsol removed to Psol release ratios determined using both methods. Table 2 shows that in the absence of EBPR the CODsol removed to Psol release ratio determined by the membrane filtration method was 28 mgCOD/mgP; when the COD was measured by the flocculation method the ratio was 2 mgCOD/mgP. The higher ratio obtained by the filtration method is attributed to the adsorption of a significant amount of colloidal material on the activated sludge flocs. No anaerobic flocCODsol removal was obtained in the absence of EBPR indicating that all of the influent colloidal matter was removed in the samples treated by Zn(OH)₂ flocculation. During periods of EBPR the overall anaerobic CODsol removed to Psol released ratio determined by membrane filtration averaged approximately 5.5 mgCOD/mgP, a value significantly higher than those reported in the literature (Wentzel *et al.*, 1985). The average anaerobic CODsol removed to Psol release ratio of 2 mgCOD/mgP, determined by the flocculation method, was in excellent agreement with the literature values reported for soluble substrates (Wentzel *et al.*, 1985; Comeau *et al.*, 1987).

CONCLUSIONS

A novel rapid physical-chemical method – the flocculation method – has been developed for the determination of the readily biodegradable portion of influent soluble COD. The flocculation method is based on two assumptions:

- 1) influent total truly soluble COD consists of a readily biodegradable fraction and a non-biodegradable fraction (IAWPRC, 1986) and;
- 2) the non-readily biodegradable influent soluble COD is

equal to the truly soluble effluent COD from an activated sludge plant treating the influent at an MCRT > 3 days (Ekama *et al.*, 1984).

The validity of the flocculation method was assessed by comparing it with the biological method developed by Ekama *et al.* (1984). Results from four domestic wastewaters demonstrated that the two methods gave virtually identical results. The Zn(OH)₂ flocculation method is fast and simple compared to the biological method especially when the wastewater is already being treated in an activated sludge system of some type. The flocculation method was used successfully to determine the quantity of truly soluble organic matter removed in the anaerobic zone of an EBPR plant.

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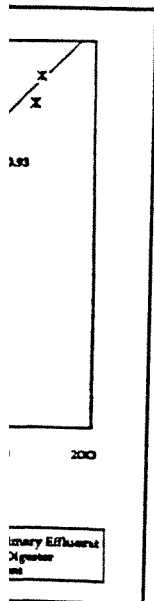
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EVALUATION OF A MODIFIED FLOCCULATION FILTRATION METHOD TO DETERMINE WASTEWATER READILY BIODEGRADABLE COD

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SUMMARY

The influent wastewater fraction readily biodegradable COD (RBCOD) is of fundamental importance in the design and operation of biological nitrogen (N), phosphorus (P) and nutrient (N & P) removal activated sludge systems. This fraction will, to a large extent, determine the magnitude of N and/or P removal that can be achieved. Both physical and biological methods have been proposed to determine the RBCOD. In this paper, the physically based flocculation filtration method of Mamais *et al.* (1993) is evaluated and refined for practical application. Results from the method are compared to two biologically based methods, the conventional square wave method (WRC, 1984) and a batch test method (Mbewe *et al.*, 1995, Wentzel *et al.*, 1995, 1999); reasonable correlation is obtained.

1. INTRODUCTION

For the design and operation of the single sludge activated sludge system for biological removal of the nutrients N and P, the influent wastewater carbonaceous material (measured in terms of the COD parameter) fraction readily biodegradable COD (RBCOD) is of fundamental importance: In biological N removal (via denitrification) systems, the readily biodegradable COD stimulates a rapid denitrification rate in the primary anoxic reactor, and the amount of denitrification achieved is closely related to the amount of RBCOD in the wastewater (WRC, 1984; Van Haandel *et al.*, 1981); in biological P removal systems, if the system is appropriately designed the magnitude of P removal achieved is directly related to the influent RBCOD concentration (Nicholls *et al.*, 1985; Wentzel *et al.*, 1990). Thus, quantifying the RBCOD fraction of wastewater is vitally important for activated sludge systems that include biological removal of nutrients. A number of experimental methods have been developed to quantify the RBCOD, both physically and biologically based.

The physical methods offer some advantage over the biological methods in that they are relatively simple and a result can be obtained in a relatively short period. For the physical methods, it has been hypothesized that the observed differences in biokinetic response of activated sludge to RBCOD and SBCOD is due to differences in molecule size - RBCOD consists of relatively small molecules that are readily transported into microbial cells whereas SBCOD comprises larger and more complex molecules that require extracellular breakdown (hydrolysis) to smaller units before uptake and utilization (Dold *et al.*, 1980). Accordingly, physical separation of the two biodegradable COD fractions on the basis of molecular size has been proposed as an approximation of the observed biokinetic division. For physical separation, filtration methods with various filter pore sizes have been used (e.g. Dold *et al.*, 1986; Lesouef *et al.*, 1992; Mamais *et al.*, 1993; Bortone *et al.*, 1994; Torrijos *et al.*, 1994). Success with the filtration methods has been closely linked to the filter pore size used - the larger the pore size, the more "particulate" material passes through the filter and the less accurate the separation between RBCOD and SBCOD. To overcome this problem, Mamais *et al.* (1993) successfully investigated flocculation of colloidal/particulate material (SBCOD) before filtration through 0.45µm filters. In this paper, the physically based method of Mamais *et al.* (1993) will be evaluated and refined for practical application. Results from the method will be compared to biologically based methods, in particular the conventional square wave method (WRC, 1984) and a batch test method (Mbewe *et al.*, 1995, Wentzel *et al.*, 1995, 1999).

Mbewe *et al.*

2. EXPERIMENTAL METHODS

Batches of raw (unsettled) wastewater collected from Borchers Quarry and Mitchells Plain Wastewater Treatment Plants (Cape Town, South Africa) were stored at 4°C and served as influent wastewater for a period of about 2 weeks. A sample was drawn from the wastewater, diluted to $500 \pm 50 \text{ mgCOD/l}$ and used for all three RBCOD testing techniques, flocculation-filtration, square wave and batch test, and as influent for a laboratory-scale completely aerobic activated sludge system (needed to determine unbiodegradable soluble COD, see below). For the flocculation-filtration method, Mamais *et al.* (1993) added zinc sulphate as flocculent to the raw wastewater and adjusted the pH to 10.5 with NaOH, this pH being the optimum for zinc sulphate flocculation. In preliminary tests it was found that the zinc sulphate flocculent recommended by Mamais *et al.* (1993) could be replaced with aluminium sulphate - this gave good flocculation and has the advantage that no pH adjustment is necessary; addition of the aluminium sulphate caused pH to decline to 6.0 to 6.3, the near optimum pH for aluminium flocculation.

For the flocculation-filtration, 1ℓ of the diluted wastewater was dosed with 10ml of stock aluminium sulphate $[\text{Al}(\text{SO}_4)_3 \cdot 15\text{H}_2\text{O}]$, Merck solution (stock at 50g/ℓ). The mixture was stirred rapidly ($\sim 200 \text{ rpm}$) for 2 minutes (rapid mix phase) and then slowly ($\sim 1 \text{ rpm}$) for 30 minutes (flocculation phase) (observations indicated that the flocculation phase could be reduced considerably to about 5-10 minutes, but this was not investigated). During the flocculation phase, flocs coalesced and settled to leave a “clear” liquid zone. A 50ml sample was drawn from the clear liquid zone and filtered through a glass fibre filter (Whatman’s GF/C) and the COD of the filtrate determined. The filtrate from the GF/C filter was then filtered through 0.45µm filter paper (Millipore HVLP) and the COD of this filtrate also determined. Both glass fibre and 0.45µm filters were used to determine if the 0.45µm filter recommended by Mamais *et al.* (1993) could be replaced with glass fibre filters to reduce costs.

In the test procedure, the filtrate derived from the influent will contain both RBCOD and unbiodegradable soluble COD (USCOD), since both COD fractions are “soluble”. Thus, it is necessary to independently determine the USCOD, to derive an estimate for RBCOD. Following the recommendations of Ekama *et al.* (1986), the USCOD was determined using the effluent from a laboratory-scale completely aerobic activated sludge system operated at 12 days sludge age (see Mbewe *et al.*, 1995 for details), tested in exactly the same manner as the influent.

The difference in COD between the flocculated-filtered influent and effluent samples gives the biodegradable “soluble” COD, which should correspond to RBCOD (Mamais *et al.*, 1993). This was evaluated by comparing the RBCOD results to those measured with the conventional square wave test (WRC, 1984; Ekama *et al.*, 1986) and a batch test (Mbewe *et al.*, 1995; Wentzel *et al.*, 1999), on the same sewage batches at the same COD concentration.

3. RESULTS

For the flocculation-filtration method, the RBCOD were calculated as the difference between the influent and effluent flocculation-filtration CODs, for both GF/C and 0.45µm filters. For the square wave and batch test methods, the RBCOD were calculated following the procedures of WRC (1984) and Wentzel *et al.* (1995, 1999) respectively; full details are given by Mbewe *et al.* (1995). For each wastewater batch tested (23 in total), from a statistical analysis the mean and standard error of the mean were determined for the four methods, see Table 1.

To compare the results derived from the flocculation-filtration method with those from the conventional square wave and the batch test methods, the different wastewater batch mean RBCOD values from 0.45µm flocculation-filtration are shown plotted against the corresponding mean values from the square wave and batch test methods in Figs 1 (a and b) respectively; a reasonable correlation was found, certainly adequate for design and operation.

To evaluate whether the 0.45µm filters recommended by Mamais *et al.* (1993) could be replaced with glass fibre filters, for the different wastewater batches the mean RBCOD concentrations from the glass fibre filtration were plotted against those from the 0.45µm filtration, see Fig 3 - very close correlation was obtained. Clearly, the 0.45µm filters can be replaced with glass fibre filters with no loss in accuracy, thereby reducing the cost.

4. CONCLUSIONS

- The zinc sulphate flocculent recommended by Mamais *et al.* (1993) can be replaced with aluminium sulphate. This has the advantage that pH adjustment after flocculent addition is not required.
- The flocculation-filtration method provides RBCOD estimates that correlate reasonably closely with those for the square wave (WRC, 1984) and batch (Mbewe *et al.*, 1995, Wentzel *et al.*, 1995, 1999) methods.

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Table 1: Wastewater (WW) source, batch number and results for RBCOD from the biological batch (Mbewe et al., 1995) and conventional square wave (WRC, 1984) tests, and the physical flocculation-filtration tests with 0.45µm and GF/C filters. Each value is the mean of a number of tests, with standard deviation of the means in () and number of tests in [].

BORCHERDS QUARRY WASTEWATER					MITCHELLS PLAIN WASTEWATER				
WW Batch No.	RBCOD AS A FRACTION OF TOTAL COD (%)				WW Batch No.	RBCOD AS A FRACTION OF TOTAL COD (%)			
	BIOLOGICAL TESTS		FLOCC-FILT. TESTS			BIOLOGICAL TESTS		FLOCC-FILT. TESTS	
	Batch Test	Square wave	0.45µm	GF/C		Batch Test	Square wave	0.45µm	GF/C
1	20 (2.0)[5]	21 (1.2)[11]	-	-	10a	22(0.5)[6]	-	20 (0.7)[9]	19 (1.4)[9]
2	11 (0.7)[5]	-	-	-	10	17 (1.2) [9]	18 (1.1)[10]	19 (1.0)[11]	18 (1.1)[11]
3	15 (0.9)[8]	15 (1.6)[5]	17 (1.9)[7]	-	11	17 (0.7)[7]	18 (1.4)[12]	17 (0.7)[11]	17 (0.9)[11]
4	20 (0.9)[8]	17 (1.6)[7]	17 (1.7)[6]	-	12	19 (1.1)[5]	19 (1.6)[9]	20 (1.5)[10]	18 (1.6)[10]
5	21 (0.9)[6]	20 (0.9)[6]	18 (1.4)[7]	19 (1.1)[7]	13	25 (0.8)[10]	20 (1.1)[12]	23 (0.7)[11]	22 (0.8)[11]
6	23 (0.4)[6]	21 (1.1)[7]	19 (1.4)[5]	19 (1.1)[6]	14	19 (0.9)[9]	17 (1.8)[10]	21 (0.6)[15]	21 (0.7)[15]
7	18 (1.0)[9]	18 (1.4)[7]	17 (0.9)[7]	17 (0.9)[6]	15	19 (1.0)[8]	-	19 (0.7)[17]	19 (0.9)[17]
8	18 (1.4)[10]	17 (0.8)[14]	14 (1.0)[5]	14 (1.0)[5]	16	22 (1.9)[7]	21 (0.6)[17]	18 (0.6)[8]	19 (1.0)[8]
9	17 (0.9)[7]	18 (1.1)[7]	18 (1.2)[8]	18 (1.3)[8]	17	27 (0.6)[10]	23 (0.7)[17]	19 (0.7)[11]	19 (1.1)[11]
					18	26 (1.5)[11]	20 (1.2)[8]	20 (0.5)[10]	19 (0.6)[10]
					19	24 (1.5)[9]	24 (1.1)[10]	25 (1.3)[5]	25 (1.4)[5]
					20	22 (1.1)[10]	21 (1.9)[6]	23 (1.1)[9]	23 (0.8)[8]
					21	20 (0.8)[8]	21 (1.1)[10]	16 (0.6)[10]	16 (0.6)[10]
					22	20 (0.5)[4]	-	-	-
					23	18 (1.3)[8]	19 (1.9)[9]	-	-

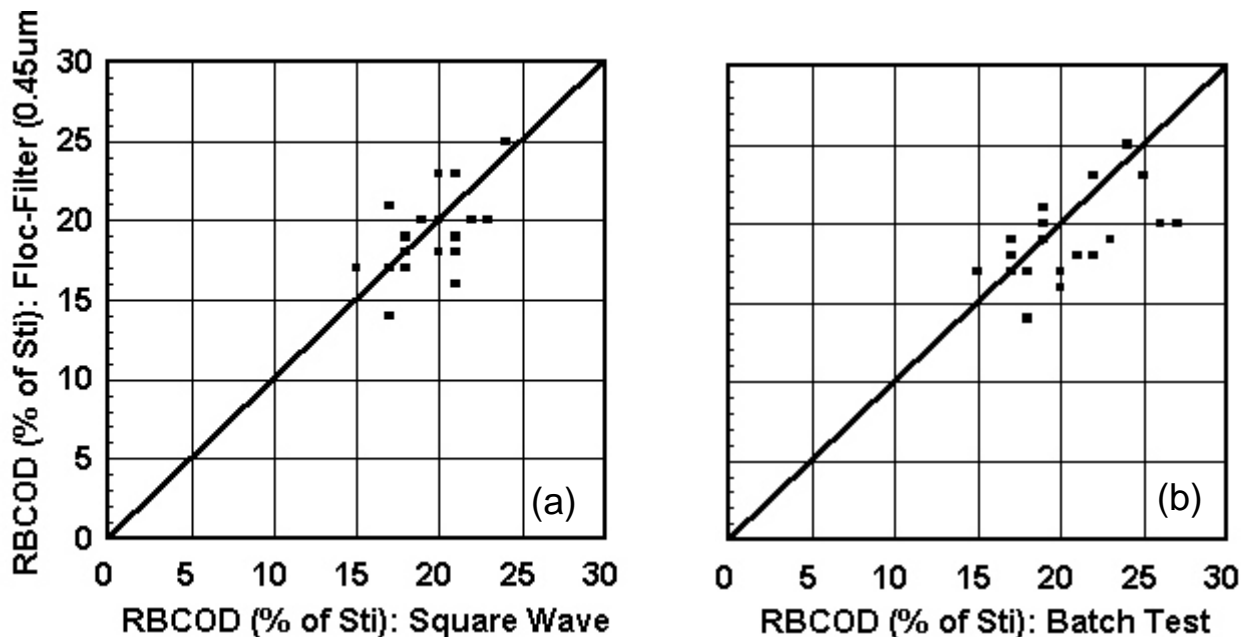


Fig 1: RBCOD as % of total COD (S_{st}) derived from 0.45µm flocculation filtration versus those from (a) square wave test (Ekama et al., 1986) and (b) batch test (Wentzel et al., 1995, 1999). Each data point is the mean of a number of tests on one batch of sewage.

- The flocculation-filtration method is relatively simple and easy to apply. However, the method does require effluent samples from a long sludge age activated sludge system, to independently determine unbiodegradable soluble COD; these may not always be available. Further, it will be difficult to match an influent grab sample to an effluent sample in a full-scale system, because the effluent will lag the influent and reflect a semi-composite sample due to the hydraulic effects in the main activated sludge basin. Accordingly, the method would be best suited to determine RBCOD on an influent composite sample or a parallel lab-scale system, and cannot be applied with certainty to determine RBCOD diurnal profiles.

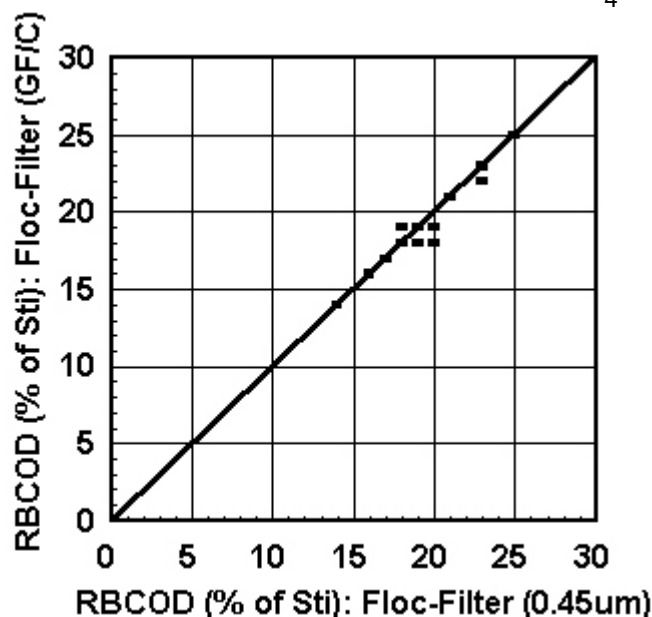


Fig 2: RBCOD derived from the glass fibre (GF/C) flocculation-filtration versus those from the 0.45µm flocculation-filtration. Each data point is the mean of a number of tests on one batch of sewage.

- In the flocculation filtration method, glass fibre and 0.45 µm filter papers give results that correlate closely. Accordingly, the 0.45µm filter paper recommended by Mamais *et al.* (1993) can be replaced by glass fibre filter paper to reduce the cost of the test procedure.

5. ACKNOWLEDGEMENTS

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